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EXHIBIT A

*TEM Debonding patent US Serial No 09/980,219
Comparison between cited references (Expancel® and
Micropertl® and TEM of the present invention*



TEM

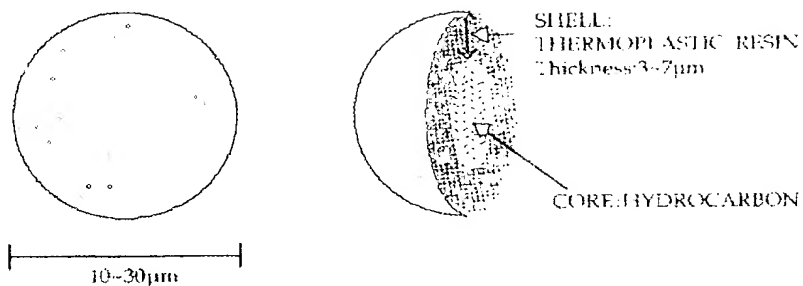


Fig. 1

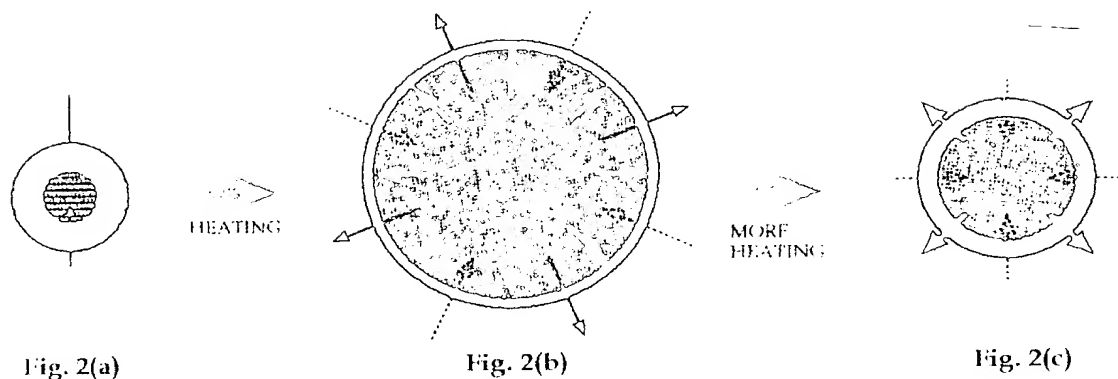


Fig. 2

Figure 1 and 2: How the TEM are composed, expand by thermal heating and is reduced in the natural cooling by shrinkage (TEM = Thermally Expandable Microspheres).

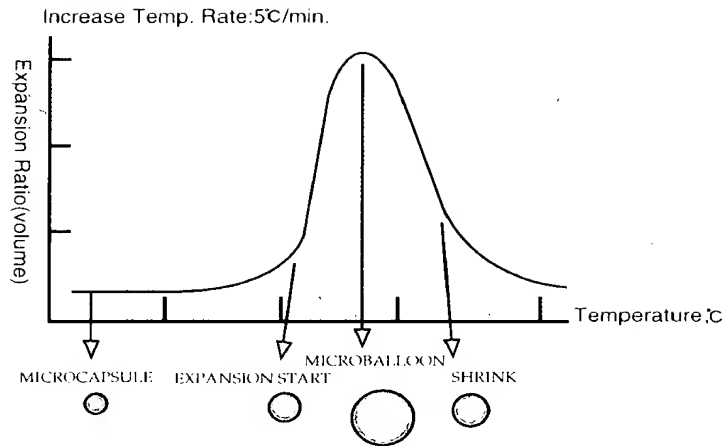


Fig. 3: Expansion ratio in function of temperature in three stages:

- heating TEM with increase temperature-time rate when no expansion occurs;
- expansion temperature range from T_i (start) (near to softening T_f of the copolymer of the shell) up to T_M (maximum expansion temperature) in the temperature-time rate usually very short;
- shrinking TEM only at T_M (expanded state) with temperature-time rate as shorter as possible to freeze in the shrinkage.

The mechanism of the three phases depends on the copolymer and thickness of the shell, hydrocarbon blowing agent, heating system as described above in the test by 1), 2), 3) and 4) points.

MAIN VARIABLES AND PARAMETERS TO BE COMPARED ON TEM TECHNOLOGIES

1) *External surface of TEM:*

- S = surface aspect, morphology and porosity; eventual coating over the shell;
 CR = chemical resistance to solvent and plastifiers of the embodying TEM matrix due to the copolymer thermoplastic: composition, structure and monomer % MO left in the shell;
 MH = moisture due to humidity and intermolecular water of copolymer in the shell.

2) *Shell:*

- h = thickness (3-7 micron) (Fig. 1 and 2);
 D_i/h_i = parameter with D_i size of TEM and h_i shell thickness both before expansion;
 D_e/h_e = parameter with D_e size and h_e thickness of TEM after expansion and after shrinkage (Fig. 2 of cooling rate);
 TCO = thermoplastic copolymer and its viscosity-temperature relationship V_{is}/T in order to predict the softening point temperature T_i when the expansion process starts with a certain time rate;
 V_i, V_c, V_{oi}, V_{oc} = volume and distribution range D_w of TEM before expansion (V_i), after expansion (V_e); V_{oi}, V_{oc} volume of the core of the TEM before and after expansion;
 D_M, D_m, V_M, V_m = D_M maximum and D_m minimum size of TEM and their volumes and distribution ranges.

3) *Hydrocarbon blowing agent as a pressure activator:*

The hydrocarbon blowing agent provides internal pressure in the cavity core of volume V_o by transforming its liquid state filling V_o , in vapor capable to raise internal pressure in the following phases of the plastic shell:

- no shell expansion (Fig. 3) up to softening temperature T_f (T_i) when the plastic shell starts flowing;
- shell expansion (Fig. 2 and 3) with thinning of the shell thickness h with diffusion (leakage) of hydrocarbon gas through the porosity of the shell up to the maximum expansion temperature T_M , in this range of temperature it is fundamental the rate of expansion $R_h = \frac{T_M - T_f}{t_c}$ with the t_c = time of expansion; the R_h depends on the heating system;
- shell shrinkage (Fig. 2 and 3) due to normal cooling after the stop of the heating system and its cooling rate $R_c = \frac{T_M - T_f}{t_c}$ with t_c the cooling time.

During the cooling time the part of hydrocarbon remains in the cavity, condenses back into liquid state (nearly 1/3 of initial hydrocarbon volume) and simultaneously through the porosity of the shell, which is solidifying, the surrounding air enters in the rest cavity and thus generates a pressure capable to keep the shell expanded in the matrix.

The complex phases of expansion and shrinkage of the shell and their rate of expansion (heating) and shrinkage (cooling) depend on hydrocarbon type H, copolymer TCO and thickness h of shell and quite important the heating source as:

- convective by hot blowing air;
- conductive between hot matrix and TEM as in an injection molding process;
- electrical power with matrix – TEM between electrically conductive plates;
- radiation power as UV, laser, sun lighting, IR special sources;
- other as ultrasonic as electromagnetic induction.

4) *Pressure development*

Pressure development by hydrocarbon blowing agent (as mentioned in point 3) as P_i and its increasing rate inside the core starting at a temperature T_i up to T_c for a time t_c of the expansion process in relation with the outside pressure constant P_e which can be atmospheric but also additionally to the volume structurally cohesive stiffness of the matrix P_c . In the cooling phase after that expanding, the role of atmospherical + cohesive pressure and their rate P_R are strongly important.

In fact the pressure mechanism (say P_e) affects the mechanism of what occurs to the P_i inside the core on which depends the stability of density reduction for the final product on the market for the applications of the available TEM.

AVAILABLE TEM AND THEIR MARKET APPLICATIONS

The available data, at the time 2/June/1999 of priority data of the WO 00/75254 A1, have been obtained from Expancel® (Akzo) and Microperl® (Matsumoto) brochure data. The available data of the available TEM is summarized below and is practically equivalent for both Expancel® and Microperl®.

1) *External surface:*

- CR = chemical resistance see Table 1 “solvent stability”;

Table 1
SOLVENT RESISTANCE

Expandability was checked after dipping samples in each solvent for 10 days at 40°C.

SOLVENT		SOLVENT	
Benzene	X	Terpene	○
Toluene	○	Perchloroethylene	○
Xylene	○	Styrene	○
MeOH	X	Ethyl acetate	X
EtOH	X	DBP	X
IPA	○	DOP	X
n - BuOH	○	Acetone	X
Ethylene glycol	○	MEK	X
n - Hexane	○	MIBK	X
cyclohexane	○	DMF	X

○ : Practically no change of expandability

X : No expansion

- W% monomer presence possible to be extracted by evaporation during heating (about 2-3%);
- MH% moisture (depending on drying system before applications).

2) *Shell:*

- h = thickness 3 to 7 but no indication each of available TEM and no indication on its distribution and thus no indication on D_i/h_i and D_e/h_e parameter (see main variables point 2);
- TCO = three thermoplastic copolymers: vinylidenechloride; acrylonitrile; metacrylic; but no mention on viscosity-temperature relationship but only the T_i (probably the T_f start of the expansion near the softening temperature of the plastic shell). No mention to the time rate phase (see Fig. 3);
- data on V_i (D_i); V_e (D_e) volume and size of TEM before and after heating expanded mechanism and only some of them with V_i (D_i) distribution diagram D_w before heating. No mention about the distribution of V_e (D_e) after the expansion and eventual shrinkage process. Only [V_i (D_i) - V_M (D_M)] difference with no time rate R_M neither R_c .

3) *Hydrocarbon blowing agent:*

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- H = iso- or n- pentane; iso- or n- butane and their mixture with no indication about volatile and vapor properties and so no pressure-time rate and no information about their performances with different heating system (even no information on thermal or absorbing radiation) basical in approaching any problem as explained in point 3.
- No information concerning the eventual difference of temperature between TEM and surrounding matrixes both under the same heating source.
- No information as about the leakage of volatile hydrocarbon through the porosity of the thinner shell nor about the rate of mechanism of $R_M = \frac{T_M - T_i}{t_c}$ nor $R_c = \frac{T_M - T_o}{t_s}$ indicated as t_c , t_s a basic parameters in the expanding and cooling mechanism of TEM (see point 3) for any heating source.

4) *Pressure development*

Pressure development range has been usually avoided for no interest in the cited references and some time avoided as it could create surface negative aspect and even porosity on the final product (shoes, paint, sealant and so on).

Only after requested data on pressure some information has been obtained which can be summarized in the well known pressure-volume-temperature equation:

$$PV = n RT \quad (1)$$

applicable only at T softening point temperature T_f of the shell with $V = V_o$ initial core volume of TEM before expansion remaining nearly constant, n molar molecular weight of blowing agent; R = gas universal constant.

No other information on details about the pressure rate P_R and the pressure remaining after the cooling phase are available to predict the consistency and stability of the expanded TEM.

The only useful pressure value was the pressure resistance of the dormant TEM before expansion (about 300 kg/cm^2), quite useful to predict the resistance to the destructive power of high speed blades or strong rate of shear during the manufacturing process especially in the injection molding technologies.

CITED REFERENCES

- PVC shoes and other products nearly 50% of density reduction;
- TPU: thermoplastic polyurethane for density reduction products;
- PE-LD; PE-HD; PS even EPDM for density reduction products;
- Others but all thermoplastic polymers manufactured by pressure injection molding where the TEM are blended first in a by pass products producing chips to be properly extruded afterwards in a mold.

The only thermosetting polymer application were in unsaturated polyester and PU in order to avoid the shrinkage after the chemical polymerization.

Other applications can be summarized in:

- Stereocopy system;
- Printing Inks;
- Antislipping (paper bay, carpentliner);
- Lightening non-woven map, wall paper, rubber parts;
- Resilient layer: car interior, underbody coating, painting, press sensitive adhesive;
- Insulating layer: Lenco paper, electric cable;
- Rigidity improving: paper, speaker corn;
- Porous cell impregnating: artificial or man made leather;
- Expandable adhesive: masking tape, sealant. The heating system is nearly in all application a blowing hot air or electrically heated contact hot plate.

In all these applications the expanding of TEM is performed during the product ion process for market as a final products and thus usually the outside pressure P_e of the surrounding ambient or matrix seems quite not strong enough to reduce the expansion mechanism of the available TEM.

Debonding technology information had not been mentioned in any of the literature for the prior art material of Expancel® and Microporl®.

DEBONDING TESTING OF AVAILABLE TEM

To simplify the debonding technology by TEM, the principle is based on blending them in an adhesive matrix or primer matrix to realize a reduction of cohesive strength in the adhesive matrix or the interface adhesive surface strength in order to detach and remove the two glued panels with particular attention to the glazing-frame direct bonding on vehicles.

The main differences between the cited references and debonding technology in regards to use of the available TEM is as follows:

1) Temperature range of expansion

The expansion of TEM has to occur by a triggering mechanism in a solid state matrix of a thermosetting structure polymer and only at a temperature below its degradation (reduced structural cohesion and toxic diffusion of some chemical elements as monomer plastifiers). Thus it has the capability for easily removing the cohesion-adhesion strength relative to structure or interface of the bead.

For PU adhesive of glazing-frame the temperature of T_M for TEM has to be $< 180-190^\circ\text{C}$ to remain consistent to leave off without char (tar, peach) formation and with no toxic diffusion of some chemical elements (monomer) of PU or its plastifiers.

To avoid unsafety debonding the temperature T_i (T_f) has to be $T_f > 110-120^\circ\text{C}$. Thus the choice of available TEM have been only them with $T_i > 120^\circ\text{C}$ and $T_M < 190^\circ\text{C}$.

2) *Aging degradation*

The TEM used in debonding operates by triggering expansion only after 10-15 years without expansion degradation as we have shown in Tab. 1 by different solvents.

As a consequence of 1) and 2) debonding performances show a large selection has early been taken to avoid available TEM with:

- $T_i < 120^\circ\text{C}$ and $T_M > 180^\circ\text{C}$;
- PU or primer with no chemical composition of the degrading solvent or PU plastifiers capable to decrease the expanding process at temperature limit of T_f (or T start) of 120°C supposed to be achieved under certain sun conditions during the aging of vehicles.

Some adhesive PU contains some plastifiers with polyol and isocyanate as MeOH and EtHO which could degrade the TEM but above all the used primers containing often MEK, Acetone solvent or DMF which are both on Table 1 able to obtain CR with available TEM. Further the catalyser of PU curing process is water, thus the % of water of the available TEM could degrade the curing process.

Both degradation processes have been tested under the suggestion of Akzo and Matsumoto with TEM in the limit temperature range of T_i up to T_M with less % of monomer MO and water MH and porosity of surface S in order to reduce the influence of CH according to their suggestion among their available TEM.

With these suggested TEM (already limited by temperature range) and some changing of curing time and primer deposition industrially used systems (very fast to reduce the possible CH) some debonding trial in the oven with temperature control of the experimental work, has given the following results: to debond by cohesion fracture the PU the % of TEM needs to be at least 20% which, a part the cost, had reduced under the limit the adhesion strength of the direct bonding.

Thus the trials have been concentrated to primer which, even first without aging test could to verify the eventual differences among the still available TEM.

The debonding by a controlled heating in a special oven was not satisfactory for the following reasons:

- Expanded available TEM compared with new debonding TEM

The thermosetting primer shows such a strong structure materials which even at temperatures up to $190-200^\circ\text{C}$, the limit to avoid the char formation of PU adhesive (impossible to control for debonding technology as it does not leave the easy removal of glazing with zero load and clean surface as it should), allows the expansion of available TEM only to those which initial size practically with more than the thickness of cured primer at interface of primer and adhesive PU.

This can be shown in the optical microscope Fig. 4 which shows that the expansion occurs only for few TEM even with 10% in the primer of 10÷15 micron thickness. This means that only some TEM with higher size D_c could expand but with % not capable for debonding effect even with 10% TEM.

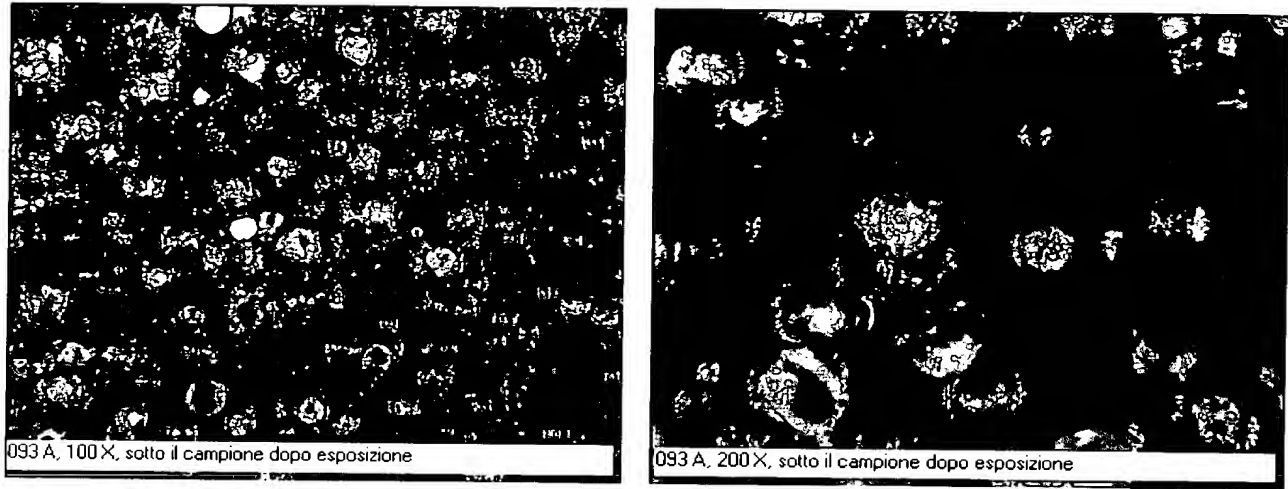


Fig. 4: 10% of special available Expancel 093/120 in a primer test at 190° for about 10 minutes.

Just to predict the real debonding efficiency of new debonding expanded TEM (specially produced in a secrecy agreement project with Akzo; the Matsumoto failed his proposed new TEM specially manufactured for De-Bonding Ltd), Fig. 5 shows the same experimental work with the same primer thickness (several tests performed), with only 3% of new TEM in weight heated by special lamp special wavelength IR spectrum in less than 3 minutes at temperature of 190÷200°C achieved only by the TEM, leaving PU at lower temperature to maintain its consistency during and after leaving the debonded surface nearly cleaned with nearly zero load off or peeling shear. The picture concerns even the debonding result after aging treatments.

The main different parameters are partly described in Fig. 5 in addition to: T_i (T_f) expansion start temperature more than 110÷120°C.

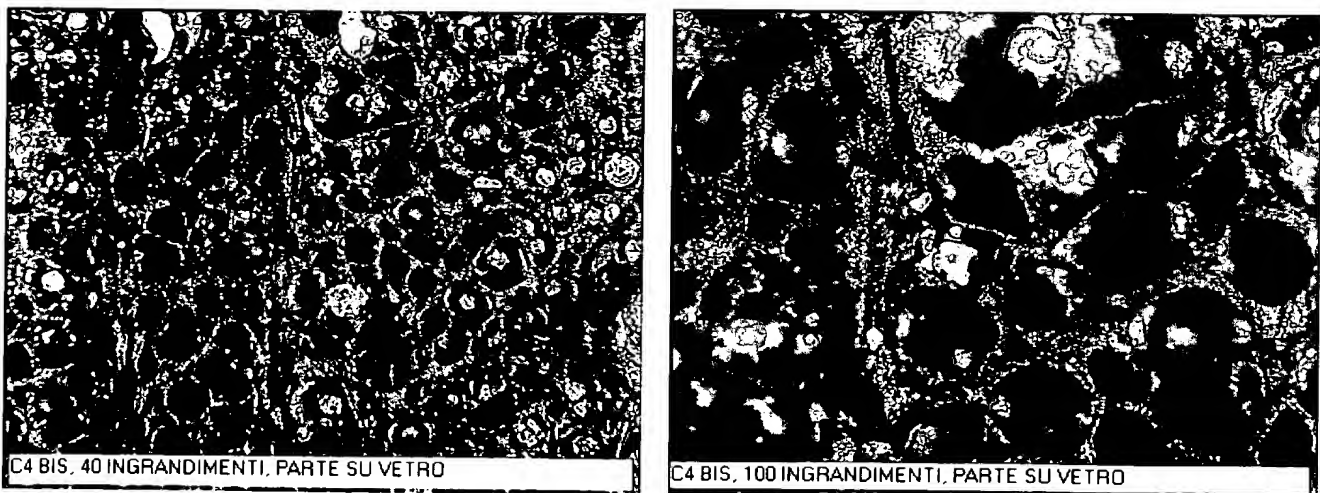


Fig. 5: Typical picture of the new expanded TEM for the real efficient debonding technology by fast heating rate R_c and fast cooling rate R_c (see Fig. 3) by IR special lamp heating source up to 190-200°C in less than 3

minutes to glazing-frame direct bonded sample with 10-15 micron thickness of well known used primer with only 3% of new TEM with special distribution initial size quite restricted with average size D_w = about 30 micron, special TCO with less % of MO, MH and porosity surface and special hydrocarbon as a blowing agent (probably octane + butane + water composition not disclosed by manufacturing). The picture is related to sample after aging treatment predicting 10 years on vehicles.

- Hydrocarbon and shell parameters differences

Hydrocarbon strongly absorbing IR special range in order to allow the TEM to achieve the maximum expanding T_M before the matrix temperature and in faster heating rate R_H and cooling rate R_c in order to leave the PU under 160°C which is the limit of consistency of PU and also with no toxic diffusion of toxic elements of PU and its plastifiers.

In addition to the hydrocarbon, new TEM with lower parameter D_i/h_i in order to get also higher relative D_c/h_c has been obtained to obtain higher expanding pressure P_{cM} in the core capable to expand more the TEM in a strong structural stiffness of primer. Thus the pressure volume temperature equation (1) becomes:

$$(P_i - P_c) V_o = n R T_f \quad (2)$$

where P_c ($P_{cM} > P_c$) is the higher simulating pressure outside the surrounding matrix even at high temperature range of expansion T_M , much more than the P_i usually present for the mentioned application by the available Expancel and Microperl.

Fig. 6 shows the expanding temperature higher than the supporting plate, even with metal comparison under a special IR lamp beam radiation in less than 20 seconds.

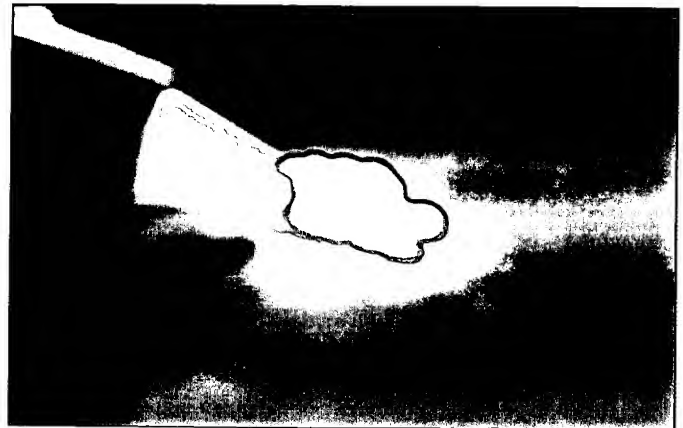
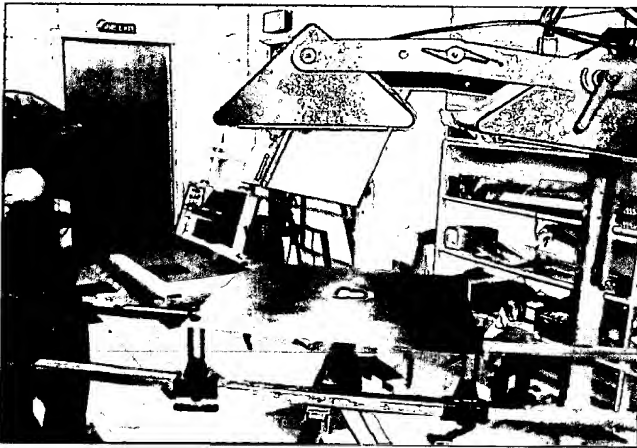




Fig. 6: The pictures verify experimentally the predicted difference between expansion T_M of new debonding TEM and the eventual surrounding adhesive temperature, under special IR radiation (power 1,5 Kw at 40 cm distance) heating. In fact the new TEM are expanding at 190°C after 17" and the temperature of the two supported plates, measured by a pyrometer (middle) show 150°C for metal (top) and even for PU adhesive on the plate. This means that the new hydrocarbon blowing agent absorbs IR giving a heating rate faster achieving 190°C when the matrix is still 150°C (40°C of difference).

- Chemical resistance after aging treatment (10-15 years)

CR resistance of more than 10% in weight of TEM shown in Fig. 6, tested for debonding with available specially suggested by manufacturing after the aging treatment (kataplasma and Dow Automotive aging test for predicting 10-15 years on the vehicles) have shown strong degradation and some toxic diffusion vapor as shown in Fig. 7.

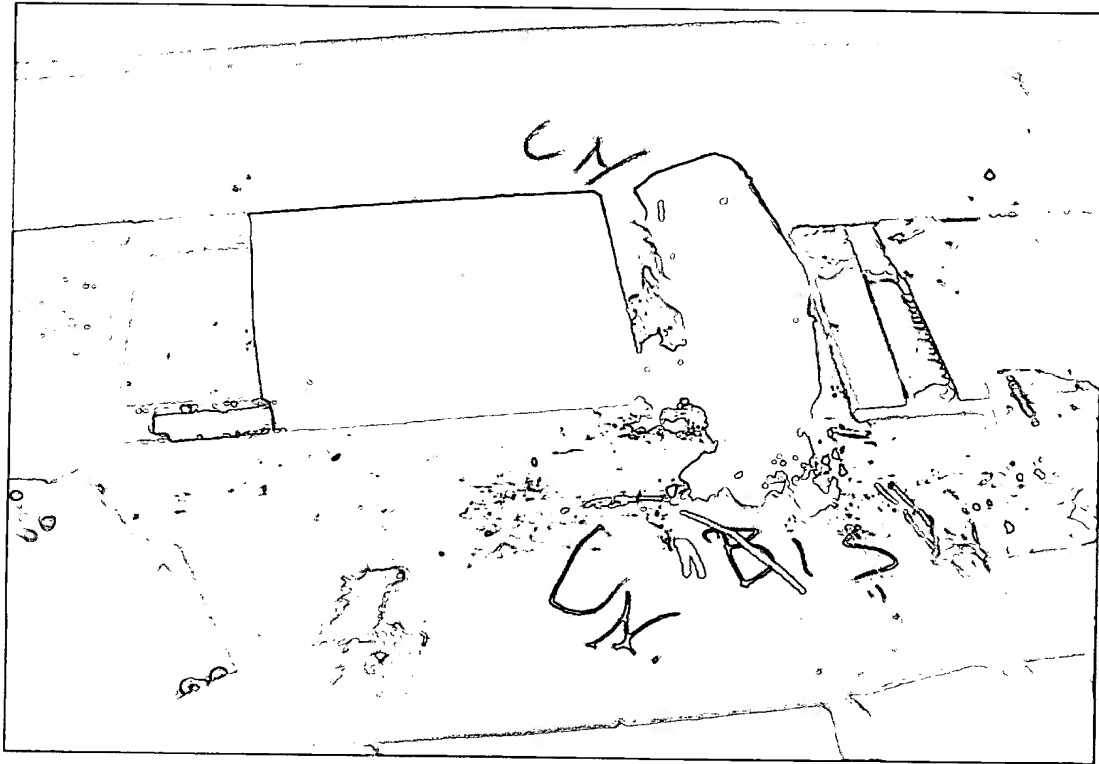


Fig. 7: IR heating time 3 minutes after the aging treatment where it is clear the fragmentation of part of the bead due to the heating IR powerful beam deterioration of the structural cohesion of the bead due to the aging Dow Automotive treatment of 14 days at 38°C/100% RH, really changes the structural consistence of the PU adhesive sealant which, at the end of 3 minutes, diffuses some toxic vapor as well. But the main fact that has to be pointed clearly out, is that the 5 or 6 minutes IR heating time has also fragmented the 14D 38°C/100% RH treated. This is a typical result of all the available aging treated TEM for debonding trials in comparison with quite good removal by new TEM.

SYNTHESIS AND CONCLUSION

Synthesis

We can now compare some of the properties of available TEM with those of new debonding necessary features which have been confirmed by experimental positive results.

Table 2 can show the wide spectrum of the verified main differences between Expancel® and Microperl® available TEM on the market as compared to those used in the present invention for debonding technology.

Table 2

Part of TEM	Variable and parameters	Available TEM features	New TEM needed features
External surface	S	Too porous	Less porosity by suitable TCO
	MP	Too high %	Less monomer %
	MH	Too high %	Less water %
	Result on CR = chemical resistance	Enough suitable for their application but no for debonding	Enough suitable for debonding especially with primer and after aging
Shell	TCO	Three thermoplastic copolymer	Only one: mixture of metacrylic + viniledene
	Thickness	3-7 micron $D_i/h_i = 7$ $D_e/h_e = 70$	Different $D_i/h_i = 4$ and $D_e/h_e = 150$ to expand with higher pressure as the external pressure in matrix is higher than that of available TEM applications
	P_i, V_i	Size and volume distribution quite wide $D_{wi} = 15\div 40$	Size D_e and volume V_e need to eliminate the lower part of the TEM distribution as only the high D_i can expand properly. $D_w = 30\div 40$ micron
	Temperature expanding range T_i up to T_M	80°C up to 220°C depending of applications which are nearly always with expanding process in the product manufacturing technology	120°C up maximum 190° to avoid aging adhesive degradation on vehicles and toxic diffusion elements of PU adhesive and primer during the heating with IR
	R_c = rate heating R_c = rate cooling	Not triggering particularly high and no fast cooling.	Triggering with high R_c and good R_c rates to avoid diffusion of toxic elements of PU which has to remain structurally consistent to be better removed and clean debonded surface.

Part of TEM	Variable and parameters	Available TEM features	New TEM needed features
Hydrocarbon	Classic blowing agent	Possible to be used for all heating systems and nearly unknown properties and different expanding and shrinkage phases	Special H octane + isobutene + water, particularly able to adsorb IR special radiation in order to get the expanding T_M faster than that of PU, leaving PU at lower temperature (about 40°C less) with still consistent solid state. The new hydrocarbon is well known for M_w = molecular weight, boiling point, density, enthalpy of vaporization and of saturation pressure, all able to control the hydrocarbon different phases of the expanding process
	Rate of diffusion through the porosity of the shell	No particularly high as the purpose is the expansion for product with lower density and other properties as acoustic and bouncing performances due to expand electrical insulation TEM	Particularly high in order to leave the condensed liquid hydrocarbon inside the core of TEM with pressure developing higher than the surrounding P_e , so increasing the efficiency of debonding (higher % of expanded TEM on the interface between primer and adhesive) (see Fig. 4 and 5)

Conclusion

By comparing the data in Table 2 the simple conclusion is that no one of the present available TEM on the market have been capable of debonding as their different new features, all together in the new TEM, have been essential for the industrial success of the debonding technology.

The available TEM are satisfactory for a lot of applications but their features, as they are still even now, are not capable for debonding technology.